

CEMENT AND LIME MANUFACTURE

XXV. No. 1

JANUARY 1952

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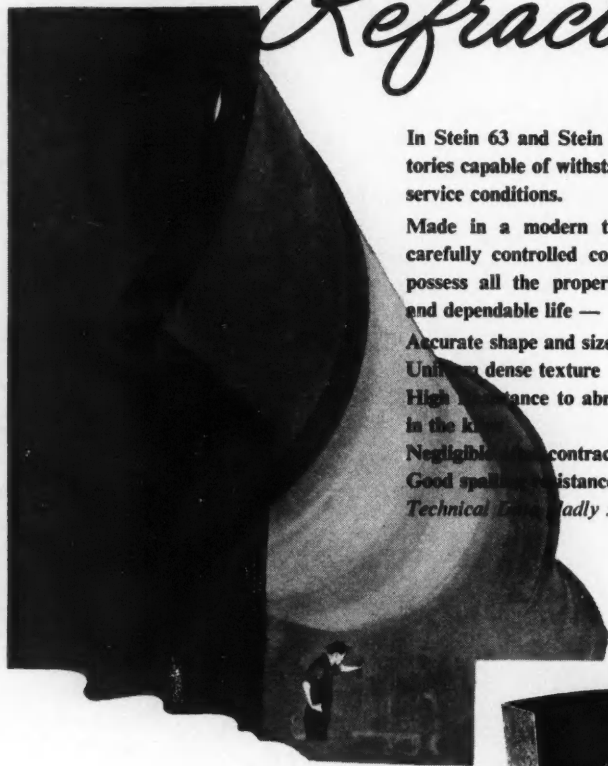
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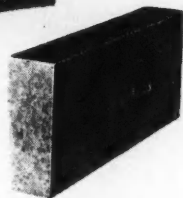
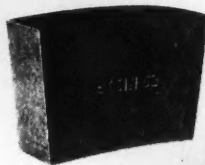
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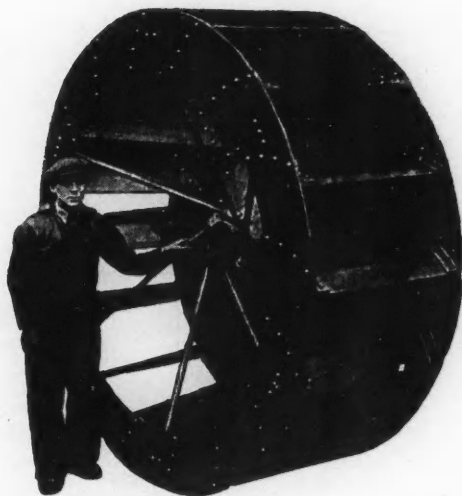
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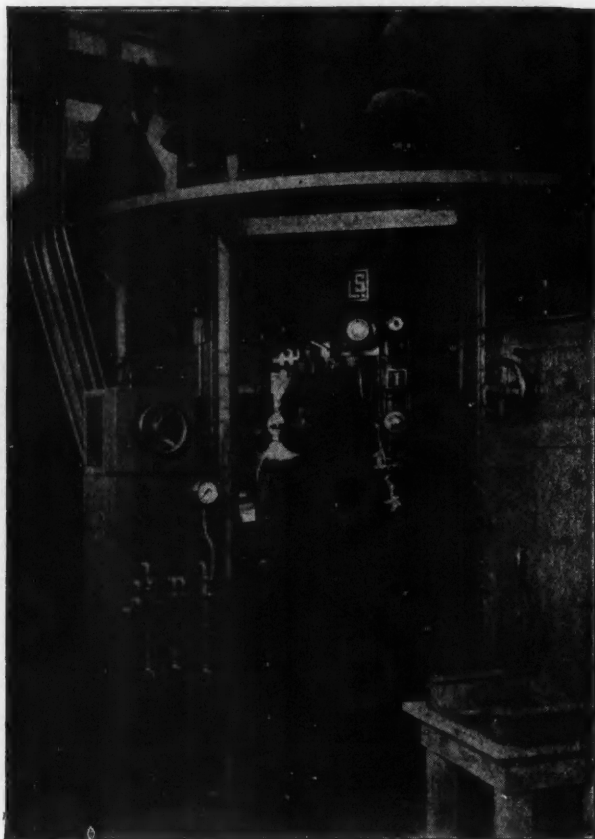
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VOLUME XXV. NUMBER 1.

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A New View of the Setting of Cement and the Nature of Concrete.

TESTS of concrete structures do not always give the same results as those derived from calculations based on the theories commonly used. To explain these variations, M. Freyssinet puts forward new ideas of the hydration of Portland cement and the constitution of concrete. He describes his theory in a paper entitled "Importance et Difficultes de la Mecanique des Betons," which was presented at the congress on prestressed concrete held in Ghent in September last. Concrete is considered to be a complex solid-liquid-vapour phase, and laws are put forward to explain shrinkage, creep, and other elastic and plastic deformations. The following is an abstract of the paper.

Hydration of Cement.

The water used for mixing mortar or concrete dissolves slightly the finely pulverised cement. Saline ions are dispersed haphazardly in the liquid and, where the solution is sufficiently rich, they form with the molecules of water relatively stable hydrated crystals. The largest crystals are less soluble and tend to absorb the smallest, and also to retard the formation of small crystals by reducing the concentration of the solution. On the other hand, a crystal can grow if ions reach its surface by passing through the liquid by channels which are proportionally more and more restricted and sinuous as the number of crystals increases, so that the ions have many chances to become grouped into new crystals where the solution is sufficiently concentrated. The average size of the crystals will therefore be smaller in proportion as the circulation of the ions becomes slower and more difficult. Therefore the number of crystals depends essentially on the rapid circulation of the ions in the paste during setting. It is evident that such circulation will be made more difficult by compression of the paste and reduction in the amount of water.

When unset cement paste is heated, setting is accelerated as the temperature is raised, but the structure of the set paste is coarser because of the formation of large crystals, and the strength is less. It is therefore necessary to limit the temperature, but if the size of the crystals can be reduced by compressing the paste, it is possible to apply heat of 100 deg. C. or more and still have a very fine structure and great strength. This theory underlies the manufacture commercially of high-strength concrete by combined heating and compression.

The constituents of concrete after a certain period of setting are as follows.

(1) Particles of various sizes of aggregate which have a high elastic modulus.
(2) Grains of undissolved cement which are surrounded partly by hydrated crystals (which hinder the continuation of the dissolution) and of which the size is between 1 mm. and 1 micron.

(3) Hydrated crystals which, at least in the presence of a large excess of water, are in the form of needles the width of which is a multiple of the diameter of the molecules of water, that is about a thousandth part of a micron.

(4) Crystals forming a close mesh the interstices of which are partly filled with water; complete compaction of this mesh is impossible because agglomeration of the crystals is only possible if material from one part of the mixture can pass to another more stable part, that is if the channels are sufficiently numerous to allow access to the surfaces of the crystals. The mesh is extremely close when the setting reaches a quasi-stable state, because this state is reached when the difficulties opposing the movement of the molecules in the channels are great. Some of the channels must contain no water since the formation of the hydrates is accompanied by shrinking.

The relative equilibrium of the mass results from local imperfect equilibrium. The grains of cement cease entering into solution only if the water in contact with them is saturated, in which state it can remain only because of the difficulty of the diffusion of the ions through the very narrow channels. For the same reason, the hydrated crystals are in equilibrium with the water surrounding them; they cease to grow because they receive more ions only rarely. But if the physical conditions change, the setting process enters an active stage. Local variation of pressure in the liquid is reflected, depending upon the sense of the variation, by local solution of the hydrates which travel towards less compressed zones, or by a decrease in the solubility of the hydrates.

It can be demonstrated that plastic extension results in a decrease of the pressure in the interstitial liquid, and therefore of a reduction in the solubility of the hydrates. By enlarging the channels, movement of the ions is favoured. There is a resumption of the process of the formation of the hydrates, which grow in the enlarged channels; this is not conjecture, since experience shows that new cracks in concrete can be healed if the distance between the faces of the fissure is not excessive and if a humid condition is maintained. Excessive mechanical compression, on the contrary, produces compression of the interstitial liquid and the solution of some of the hydrates.

These actions are of great importance, as they affect the ratio of the resistance to compression to the resistance to tension, which is about 10 to 1 for concrete.

Although for some brittle materials the tensile resistance is the basis of the working conditions, in reinforced concrete structures the concrete near the reinforcement is subjected to a tension generally sufficient to cause cracks even under partial load. Under the greatest working load for which a reinforced concrete member is designed, the concrete may be more severely acted upon, and as a result the elongation is increased (but not the stress which is already the maximum value to cause cracking). These effects are only possible because concrete is able to adapt itself to circumstances if the time and conditions necessary for their production are available.

Not only the plastic deformation, but all deformations of concrete depend on the properties of the spaces in the mesh formed by the hydrated crystals. The spaces are filled partly by water and partly by vapour, these phases being separated by a meniscus subjected to a constant stress, the capillary tension, which causes a hydrostatic tension p in the liquid phase, balanced by a compression in the solid phase. It can be shown that $p = \frac{A}{2D}$ if D is a function of the dimensions of the interstices between the crystals and A is a constant which can be determined experimentally. From thermodynamical laws it can also be shown that

$$\Sigma = \frac{1}{e^{\left(\frac{2A}{1300D}\right)}}$$

in which Σ is the hygrometric state of the atmosphere above the liquid. From these laws the following can be deduced.

(1) If a concrete contains a certain quantity of water, the water fills the superficial spaces the value of D of which is a certain value because, if it were otherwise, there would be displacement of water from wetter zones to others across the liquid and vapour phases; therefore uniformity of D is a limiting state towards which the system tends.

(2) For this special value of D there is a certain value of Σ_1 which characterises the hygrometric state of the concrete. There is, in effect, equilibrium and a total absence of exchanges between a concrete and an atmosphere of the same hygrometric state.

(3) If the hygrometric state of the concrete which results from the equilibrium value of D is greater or less than that of the atmosphere, there would be a transference of molecules from the water to the atmosphere or vice versa, the pressure p increasing when Σ decreases, or vice versa.

If v is the volume of the parts of the channels which are full of water (that is the volume of the liquid phase of the concrete), the total pressure is pv , which is a triple stress causing a linear deformation $\frac{pv}{E}$, where E is the shortening produced by a triple stress of unity. This deformation is the well-known phenomenon of shrinking of cement paste upon setting, which is simply the deformation due to a triple compression.

In passing from a hygrometric state Σ to a lower state Σ_1 , v decreases and p increases. The fact that shrinkage actually occurs shows that pv increases as p increases. The primary effect of compression is a reduction of the volume of the channels, and so v diminishes further.

Among the many phenomena accompanying loss of water are movement of water and vapour and the destruction and formation of hydrated crystals, but the speed of the phenomena diminishes as the intensity of their initial cause, that is the difference $\Sigma - \Sigma_1$, decreases.

The deferred deformation due to compression can be studied by considering a concrete that has been progressively dried to a hygrometric state Σ , which is assumed to be constant throughout the mass and equal to that of the surrounding air. If the concrete is subjected to a permanent compression, water is displaced and occupies interstices having a higher value of D . Without altering v , Σ becomes Σ_1 , p becomes p_1 , and pv becomes p_1v , and the concrete is submitted to an expansive cause. Therefore the concrete undergoes instantaneously a deformation which is only a small fraction of that which it would undergo if no change of its hygrometric state occurred due to the compression. This instantaneous deformation depends not only on permanent characteristics but on other causes, notably the initial shrinkage (that is Σ), p , and v which obtain at the moment it is compressed. Numerical expression of these phenomena is difficult because they depend on the variation of v with D , that is on the geometrical properties of the spaces in the mesh of hydrates, besides being modified by the compression. A further complication is that movement of the water introduces resistances due to viscosity. It is clear, however, that concrete of a hygrometric state greater than that of the air tends by evaporation to become in hygrometrical equilibrium with the air, and therefore undergoes further shrinking, which constitutes a part at least of the deferred deformation.

The Behaviour of Concrete.

In addition to phenomena related to the nature of hydrated cement there are those due to the heterogeneous physical nature of concrete.

If two opposing compressive forces act on two irregular-shaped pieces of coarse aggregate, sliding or shearing action is exerted on the mortar between the two pieces. The deformation due to the shearing action results in fracture of the cementitious hydrates in certain zones. The resistance of the concrete is not altered even if the forces are applied repeatedly (assuming the forces are not sufficient to disintegrate the concrete). The broken crystals must therefore be replaced instantly by others. All deformations, however small, therefore bring into play the mechanism of setting, that is the process of autogenous healing. The deformations are, however, due to inequalities, and are caused when the applied forces exceed the passive resistances. When the movement commences the balance is suddenly upset and the passive resistances suddenly decrease, so that instead of a continuous and reversible deformation, like an elastic deformation, there is a series of irreversible deformations alternating with halts in the movement. This phenomenon can be observed.

It is apparent that when forces are applied to concrete it reacts to the extent to which the forces, added to the internal stresses of the concrete, can overcome the resistance to the movement. The result varies according to whether the action leads to an increase in volume (tending to increase the porosity) or to consolidation, and also depends on the previous unknown state of the internal stresses.

When concrete dries it does so first at the surface; the interior loses its water only slowly and tends to retain its original length. Hence large tensile stresses occur at the surface causing it to stretch. When the drying reaches the interior, the stretched surface compels the interior to stretch. If therefore a free block of concrete undergoes cycles of wetting and drying (or thermal variations) there are produced as many small elongations as cycles. This phenomenon also has been observed, and it can be shown that if the concrete is compressed by being sufficiently loaded it ceases to elongate and appears to shorten. The factors comprising the deformation of concrete subjected to compression are as follows.

First, there are in unloaded concrete initial deformations which depend on the curing of the concrete and which are difficult to estimate. On application of the load there is immediately a reversible elastic deformation which is followed by a deferred elastic deformation, which is only a fraction of the first elastic deformation and is masked temporarily by the re-establishment of the hygrometric state of the concrete. There is also a plastic deformation which is the ordinary very slow process of crystallisation and dissociation of the hydrates which constitutes the mechanism of setting. The applied load increases the chances of those phenomena which result in a decrease in volume, and the effect known as creep is produced. Some investigators consider that the whole of the deferred deformation is non-reversible creep, but the author is of opinion that it is the sum of a creep (that is the tendency of a system towards maximum stability and regarding the reversibility of which the author has no knowledge) and a deferred elastic deformation (deferred by the wetting of the concrete due to pressure) which is entirely reversible.

Test for Workability of Lime Putty.

THE report of the Building Research Board for the year 1950 (London: H.M. Stationery Office. Price 4s.) states that high-speed cinematography has been applied to an examination of the action of the flow-table, an instrument used to evaluate the ease of working of lime putties and adopted in the current British Standard for Building Limes (B.S. 890). The flow-table measures workability under complex conditions of stress and strain. The present examination shows that the primary factors involved are the yield value, the resistance to plastic deformation by stresses exceeding the yield value, and the elasticity. Under the conditions of the test an increase in the workability figure given by the flow-table is associated with an increased yield value and a decreased resistance to plastic deformation. All the limes tested so far have shown appreciable elastic recovery.

Revised Canadian Specifications for Portland Cement.

THE requirements of the revised Canadian standards for ordinary, rapid-hardening, and sulphate-resistant Portland cements (CSA.A5-1951) are summarised in the following.

The maximum amount of MgO is 5 per cent. in ordinary and rapid-hardening cements and 4 per cent. in sulphate-resistant cement. If the amount of $3CaO.Al_2O_3$ is not less than 9 per cent. the maximum amount of SO_3 is 3 per cent. in ordinary and $3\frac{1}{2}$ per cent. in rapid-hardening cement. If the amount of C_3A is less than 9 per cent., the maximum amount of SO_3 is $2\frac{1}{2}$ per cent. in ordinary and sulphate-resistant cements and 3 per cent. in rapid-hardening cement. The amount of C_3A must not exceed 4 per cent. in sulphate-resistant cement. The maximum amount of insoluble residue is 0.7 per cent. and the loss on ignition 3 per cent. for all types of cement, and the amount of added material other than water and $CaSO_4$ must not exceed 1 per cent.

The initial setting time of cement paste of normal consistency must not be less than 45 minutes if tested by the Vicat apparatus or 60 minutes by the Gillmore apparatus. The final setting time by either method must not exceed 8 hours. The expansion of cement paste of normal consistency, when tested in an autoclave, must not exceed 1 per cent. A paste of normal consistency is such that the 1-cm. plunger of the Vicat apparatus penetrates 10 mm. in 30 seconds. The maximum expansion of sulphate-resistant cement subjected to a mixed sulphate test is 0.2 per cent.

The tensile strength of 2.75 : 1 mortar and the compressive strength of 3 : 1 mortar must not be less than the following :

		Tensile Strength (lb. per square inch)				Compressive strength (lb. per square inch)				
Age of specimen (days)	1	3	7	28 (wet storage)	1	3	7	28 (wet storage)
Ordinary cement	—	200	275	350	—	900	1800	3000
Rapid-hardening cement	275	375	—	—	1250	2500	—	—
Sulphate-resistant cement	—	150	250	325	—	750	1500	2800

The mortars for the strength tests are made with Ottawa sand graded from sieves No. 20 to No. 30 for the tensile test, and No. 100 to No. 16 for the compressive test. The percentage of water in the mortar for the tensile test is 6.5 plus one-sixth of the percentage required in the paste of normal consistency. The moulds are filled in one layer which is compressed twelve times by the thumbs.

The consistency of the mortar for the compressive test is such that the spread on the flow table is between 100 per cent. and 115 per cent., the approximate

amount of water to give this result being 55 per cent. The mould is filled in two layers each of which is tamped 32 times.

The fineness of ordinary and sulphate-resistant cement must be such that the residue on a sieve having 200 meshes per inch does not exceed 18 per cent. No fineness test is specified for rapid-hardening cement.

The specification (price 75 cents) is issued by the Canadian Standards Association, Ottawa.

The Reaction of Portland Cement with Opal.

A series of papers by Mr. H. E. Vivian and Mr. C. E. S. Davis, of the Commonwealth Scientific and Industrial Research Organisation, on the reaction of Portland cement and opal found in aggregates in some parts of the world have been reprinted and issued by the Organisation (Lorimer Street, Fishermen's Bend, Victoria, Australia. No price stated). The papers are entitled "The Effect of Hydroxyl Ions on the Reaction of Opal," "Some Effects of Temperature on Mortar Expansion," and "The Effect of Soda Content and of Cooling Rate of Portland Cement Clinker on its Reaction with Opal in Mortar."

Mr. Vivian states that there is strong evidence indicating that the attack of opal is caused by hydroxyl ions. The negative hydroxyl ions combine with the silica or reactive aggregate to form complexes with which the positive ions are associated. Although the positive ions do not initiate the silica reaction they exert a great effect on the properties of the reaction product and consequently modify the swelling of the reaction product and the expansion of the mortar. The positive ion determines very largely the nature and properties of the reaction product. Both sodium hydroxide and tetramethyl ammonium hydroxide produce clear gels when they react with silica in the presence of water. Since these gels appear to be similar the sodium and tetramethyl ammonium ions have similar effects on the complexes. The positive calcium ion, however, in combination with silica produces a white precipitate (a calcium hydroxide-silica complex) that has distinctly different properties from those of the clear gels. This calcium hydroxide-silica complex cannot swell or cause mortar expansion. Consequently it is apparent that mortar expansion cannot be caused by reaction between calcium hydroxide and reactive aggregate. There are very marked differences between the expansions of mortars that contain sodium hydroxide and those that contain tetramethyl ammonium hydroxide. The diffusion rates in mortar of sodium (and probably potassium) ions on the one hand and of tetramethyl ammonium ions on the other appear to be very different, and these differences must affect the diffusion rates of the hydroxyl ions in the solution. As a result of these differences the rate of reaction of the aggregate and consequently the rate of mortar expansion must be affected. It also seems reasonable to assume that tetramethyl ammonium hydroxide would penetrate reactive aggregate particles more slowly than sodium hydroxide does. Mortar expansion, however, depends not only on the quantity

but also on the rigidity of the reaction product. The rigidity at any instant is known to depend on the number of ions of the reactant and on the water content of the reaction product. In addition, rigidity may be affected by differences in degree of hydration of the positive ions. Although there are considerable differences in behaviour between tetramethyl ammonium hydroxide and sodium hydroxide these are in degree, not in type. When the behaviour of these compounds is contrasted with that of trimethylamine and alkali metal salts it can be seen that, in reactions with silica, the hydroxyl ion must be considered to be the reacting ion.

Mr. Vivian found that elevated storage temperatures increase the rates of aggregate reaction and of water absorption. At the same time, however, the quantity of reaction product that is sufficiently rigid to exert a directional swelling pressure on the mortar, and thus widen cracks, is rapidly decreased. Thus, although elevated temperatures increase the initial rate of mortar expansion, they decrease total expansion.

In Mr. Davis's investigations, twenty-one laboratory-prepared Portland cement clinkers, which contained from 0.03 to 2 per cent. sodium oxide, were examined for reaction and expansion in mortars made with opaline aggregate. Clinkers containing as little as 0.21 per cent. sodium oxide caused reaction but not expansion. Those containing 0.35 per cent. or more of sodium oxide caused delayed expansion. Mortars made with slowly-cooled clinker generally expanded more rapidly than those made with quickly-cooled clinker of the same soda content. In particular, the investigation supported the view that, when a constant proportion of opal is present, the alkali content of the clinker or cement is the main factor which determines the extent of aggregate reaction and of delayed mortar expansion. Although alkali may be made less readily available by rapid cooling of the clinker during manufacture, this does not prevent eventual expansion of mortar.

Unset Cement Resistant to Moisture.

It is reported that a new type of cement whose properties are unaffected by prolonged storage in moist conditions, or even by brief contact with water, is being used in Russia. It is stated that when the cement is mixed with aggregate the water-resistant film is removed and the cement sets and hardens in a normal manner.

It is also reported from Moscow that three Russian professors, named Tung, Skramtayer and Mikhailor, have been awarded Stalin Prizes for inventing expanding cement, alumina cement and the "hydrophobic" cement mentioned above, which are claimed to be "a great success of Soviet science and technics."

Proposed New Cement Works in Rhodesia.

It is reported that the Rhodesian Cement Company has taken an option on a deposit of limestone near Fort Victoria, where a new cement works will be built if the results of investigations are satisfactory.

The Extension of a Cement Works in Holland.

THE extension of the works of N.V. Eerste Nederlandsche Cement Industrie at Maastricht, Holland, which was put into operation in September last, has increased the annual output of the works by 250,000 tons, making the total output 630,000 tons. The following is abstracted from a description of the works published in the Netherlands journal "Cement" for September-October, 1951.

The plan of the new works is shown in *Fig. 1*. Marl is brought by rail to the old works, where it is mixed with clay, pyrites and water. The mixture is finely

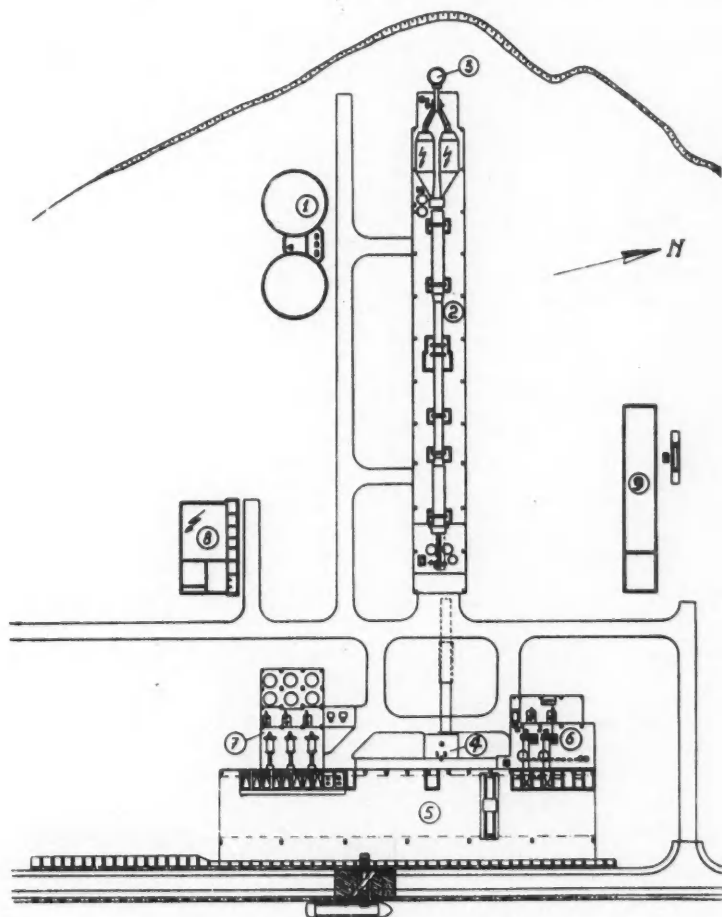


Fig. 1.—Plan of Extension.

ground in ball-mills and the slurry is pumped to the slurry tanks (1) at the new works, where it is more intimately mixed by rotating arms and injected air. The slurry is then pumped to the rotary kiln (2), which is 391 ft. long (*Fig. 2*) and 11 ft. 6 in. and 13 ft. 9 in. diameter. The kiln dust is collected electrostatically and the filtered gases pass up the chimney (3). The clinker is crushed at (4) and taken to the covered store (5). The clinker is collected from the store (*Fig. 3*) by an overhead travelling crane, gypsum is added, and the mixture is finely ground in ball-mills (6), and then pumped to storage silos at the old works.

Coal is brought to the works by boat and unloaded by grab-crane at the new quay in front of the store (*Fig. 4*) and deposited in the store. It is dried and ground

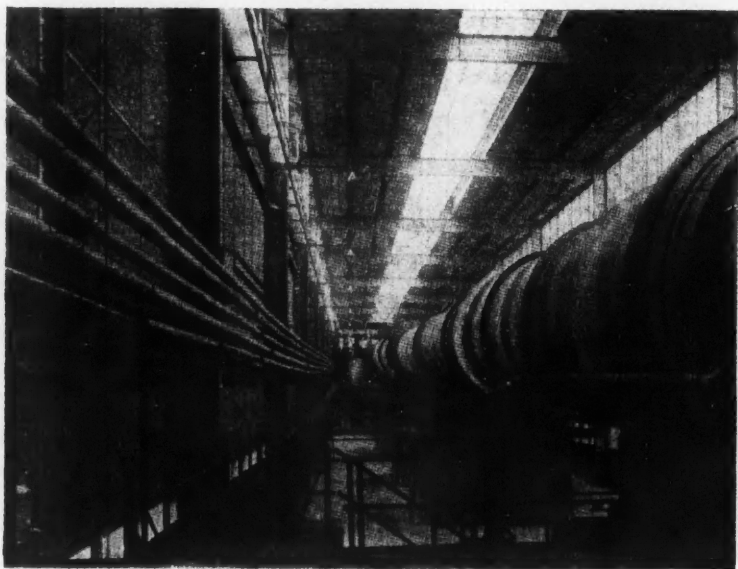


Fig. 2.—New 391 ft. Kiln.

in the building at (7), and pumped through pipes to the four kilns at the old works as well as to the new kiln. The sub-station (8) is a reinforced concrete building 114 ft. long and 74 ft. wide and has a capacity of 10,000 kW. High-tension electricity is transformed to the working voltages of 3,000 volts and 500. There is also a stores (9). Some of the cement from this works is despatched loose in barges (*Fig. 5*).

The covered store (5), which is 475 ft. long, is an important part of the new works. Five or six different designs were considered, and of these a building with a concrete vaulted shell roof was the most economical. To make the range of the quay-side crane as large as possible, the columns on the front of the store are at about 80 ft. centres. Material can be piled in the store to a height of 46 ft.,

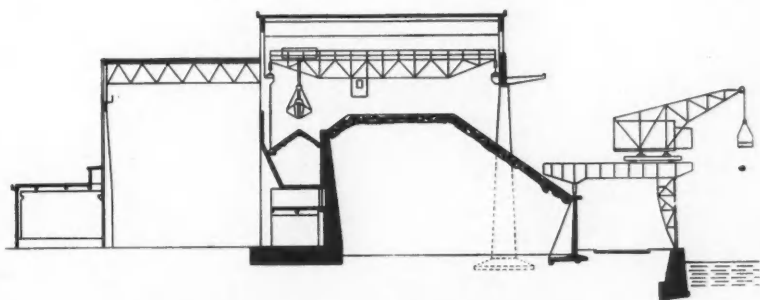


Fig. 3.—Cross Section of Quay and New Store.

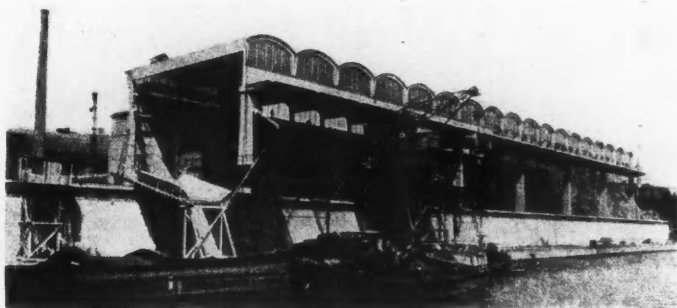


Fig. 4.—New Store.

and the capacity is 50,000 tons of clinker, 10,000 tons of coal, 10,000 tons of stone, and 15,000 tons of gypsum.

The building in which the coal is dried and ground is more than 100 ft. high, and is a reinforced concrete frame structure with walls of steel lattice with brick panels. The separation of the supporting and filling elements of the walls was an advantage during assembly of the machines, as they could be placed from outside the building before the steel lattice and brickwork were constructed. In the event of a future extension of the building, the walls can be removed without interfering with the structural members.

Pipes for pulverised coal, kiln dust, cement, slurry, and compressed air are in the open, an arrangement that has the advantage of easy control and accessibility for repairs. The pipes are carried on precast reinforced concrete posts which were made at the works.

The kiln was brought in sections by boat to the new quay and conveyed to the kiln house on lorries. The kiln house, which is of structural steel, was not erected until the kiln was installed, and is 636 ft. long and 64 ft. high. Allowance is made for a kiln house of the same size to be built on each side and, for this



Fig. 5.—Loading Loose Cement into Barges.

reason, natural lighting is provided from the roof. Ventilation is installed to carry off the heat radiated from the kiln.

The buildings, machinery, and electrical installation were planned by the company, but the elevations of the buildings were the subject of an architectural competition. Work on the site started in November 1949, when the construction of the quay wall and concrete roads was commenced. The total cost of the new works was about 15 million guilders, of which 40 per cent. was for the buildings, 45 per cent. for the mechanical plant, and 15 per cent. for the electrical installation.

Cement Factory for Jordan.

It is reported that the Jordan Government has contracted with a German firm for the supply of a dry-process cement plant with a vertical kiln. It is expected that the works will be in operation early in the year 1953, and will save the importation of cement at a cost of about £500,000 a year.

Aqueous Cementitious Systems Containing Lime and Alumina.

A BULLETIN entitled "Aqueous Cementitious Systems Containing Lime and Alumina," by Mr. Harold H. Steinour (published by the Portland Cement Association, Chicago) reviews the literature on the chemical composition of the aluminous products resulting from the hydration of Portland cement, and the nature of the setting process. The review is primarily intended as a background for further research since the problem of determining the aluminous products of hydration is far from being solved. The work is not confined to the reactions of Portland cement but is a general study of the simpler systems including alkalis, and therefore deals with the hydration of other cements such as high-alumina cement. The review contains an extensive bibliography. A summary is given in the following. Generally the statements made apply to results obtained at room temperature.

The more commonly observed calcium-aluminate hydrates are hexagonal or pseudo-hexagonal $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot (7 \text{ to } 9)\text{H}_2\text{O}$, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot (10 \text{ to } 12)\text{H}_2\text{O}$ and $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot (12 \text{ to } 14)\text{H}_2\text{O}$, and cubic $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$. Other calcium-aluminate hydrates that have been reported are $\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 10\text{H}_2\text{O}$, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot (18 \text{ to } 21)\text{H}_2\text{O}$, $5\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 34\text{H}_2\text{O}$, and $6\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 33\text{H}_2\text{O}$.

Phase relations in the system $\text{CaO}-\text{Al}_2\text{O}_3-\text{H}_2\text{O}$ have been materially clarified by several investigators^{1, 2}. Metastable relationships have not been completely worked out. Metastable forms often tend to be highly persistent.

At much above room temperature, the only stable calcium-aluminate hydrate in the system $\text{CaO}-\text{Al}_2\text{O}_3-\text{H}_2\text{O}$ is apparently cubic $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$, and is still probably the only stable hydrate at 20 deg. to 25 deg. C., although, in the small range of concentrations from 1.08 g. of CaO per litre to saturation with $\text{Ca}(\text{OH})_2$ (about 1.15 g. of CaO per litre) the tetracalcium-aluminate hydrate is highly persistent, and has a low solubility approaching that of the cubic tricalcium compound. Transformation of $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot (12 \text{ to } 14)\text{H}_2\text{O}$ to $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$ in saturated limewater has been reported, but more evidence is desirable to demonstrate thoroughly the relative stabilities.

The metastabilities of the dicalcium- and tetracalcium-aluminate hydrates have been partially established² and a solubility curve has been produced for hydrates that vary in composition from about $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{aq.}$ to $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{aq.}$ X-ray analysis indicated that the compositions were intimate mixtures of the two hydrates rather than solid solutions. It was concluded that $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot (10 \text{ to } 12)\text{H}_2\text{O}$ is such a mixture. Hydrates of varying ratios of $\text{CaO} : \text{Al}_2\text{O}_3$ which appear to be homogeneous have also been reported.

The calcium-aluminates of lower lime content, $3\text{CaO} \cdot 5\text{Al}_2\text{O}_3$, $\text{CaO} \cdot \text{Al}_2\text{O}_3$, and $5\text{CaO} \cdot 3\text{Al}_2\text{O}_3$, and high-alumina cement in which they are found, react readily with water and tend apparently to establish a metastable equilibrium involving hydrous alumina gel and dicalcium-aluminate hydrate. Metastable monocalcium-aluminate hydrate may also form.

Tricalcium-aluminate, $3\text{CaO} \cdot \text{Al}_2\text{O}_3$, reacts especially actively with water and forms hexagonal plate crystals formerly thought to be $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot (10 \text{ to } 12)\text{H}_2\text{O}$. Investigation² indicates, however, that the hexagonal phase thus formed will have differing ratios of $\text{CaO} : \text{Al}_2\text{O}_3$ depending on the proportion of water used. This is confirmed by other experiments. The crystals tend, of course, to transform to the stable $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$.

In saturated limewater, the anhydrous calcium-aluminates should apparently initially give hexagonal or pseudo-hexagonal $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{aq.}$, which may transform eventually to cubic $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$. Experiments with $3\text{CaO} \cdot \text{Al}_2\text{O}_3$ have partially verified these predictions from phase studies. When the anhydrous calcium aluminates are made with limited amounts of water, as in pastes comparable to practical cement pastes, visible crystal formation is not pronounced and gelatinous products tend to dominate.

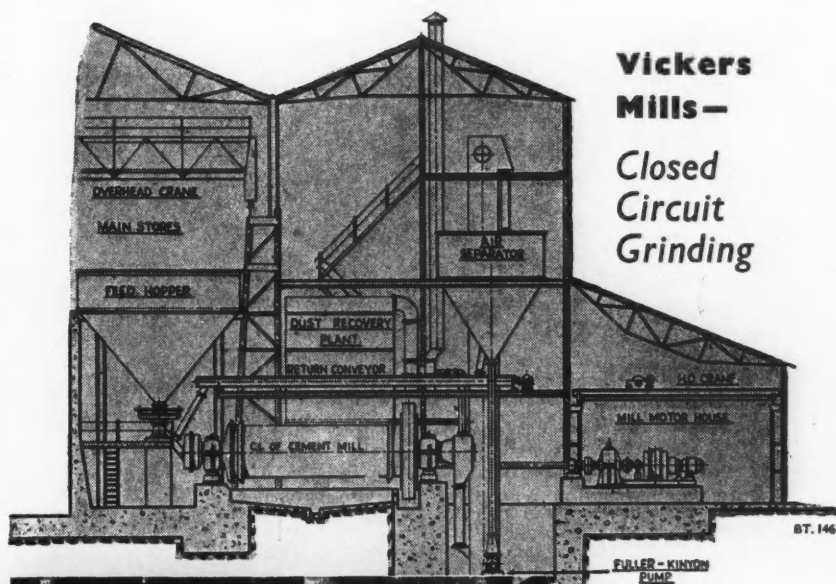
The system $\text{CaO} - \text{Al}_2\text{O}_3 - \text{CaSO}_4 - \text{H}_2\text{O}$ embraces the calcium-sulphoaluminate of high sulphate content, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$ as a stable compound^{3,4} and the sulphoaluminate of low sulphate content, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$, as a metastable product which forms a complex series of solid solutions with the hexagonal or pseudo-hexagonal calcium-aluminate hydrates. A series of solid solutions between $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$ and $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 12\text{H}_2\text{O}$ was reported earlier⁵.

It has been found^{6,7} that the addition of K_2O or Na_2O to the system $\text{CaO} - \text{Al}_2\text{O}_3 - \text{CaSO}_4 - \text{H}_2\text{O}$ by an amount equivalent to 1 per cent. KOH or NaOH modifies the phase regions but produces no change in the stable solids formed. It has been found⁵, however, that solid solutions of $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$ and $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 13\text{H}_2\text{O}$ persisted throughout test periods up to 18 months in solutions containing 1 per cent. and more of NaOH . It has also been found⁸ that there is no conversion of $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$ during an exposure for sixty days to a 3-per cent. NaOH solution.

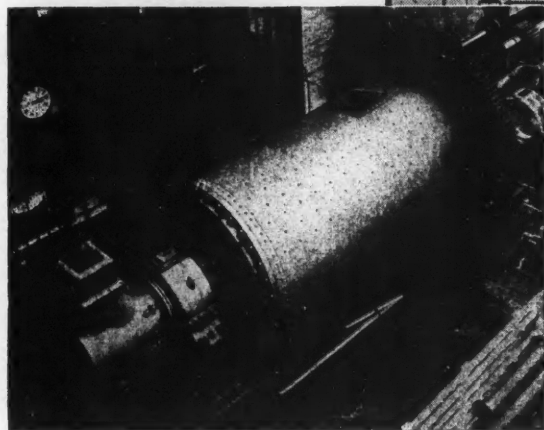
Cubic $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$ is capable of forming solid solutions with $3\text{CaO} \cdot \text{Fe}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$, and sometimes does so when $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$ (a member of a series of solid solutions represented in Portland cement) reacts with water. Solid solutions between $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{aq.}$ and $4\text{CaO} \cdot \text{Fe}_2\text{O}_3 \cdot \text{aq.}$ have also been reported and may be formed when $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$ is hydrated in saturated limewater. Limited solid solution between $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot \text{aq.}$ and $3\text{CaO} \cdot \text{Fe}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot \text{aq.}$ has been reported. Partial substitution of Fe_2O_3 for Al_2O_3 may perhaps occur in aluminous compounds formed during the hydration of Portland cement, a possibility that must be kept in mind as qualifying some of the statements that follow.

No direct evidence has yet been produced of the formation, during the reaction of Portland cement and water at room temperature, of a calcium silico-aluminate.

Reaction of Portland cement clinker with a limited amount of water quickly produces a saturated solution of $\text{Ca}(\text{OH})_2$ and should apparently cause precipitation of $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot (12 \text{ to } 14)\text{H}_2\text{O}$. Whether this product is ordinarily obtained in a clinker-water paste has not been thoroughly demonstrated, however, for crystal formation in such pastes is in general difficult to establish. In experiments



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in which more water has been used, formation of $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot (10 \text{ to } 12)\text{H}_2\text{O}$ has been reported. Eventual transformation of the initial product to cubic $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$ is a possibility, although, direct evidence for this compound in aged clinker or cement paste is also meagre.

Portland cement, which contains a small percentage of gypsum to retard setting, reacts with water with initial formation of a calcium sulphoaluminate, but whether this compound is, in general, a high-sulphate or low-sulphate form is still not thoroughly established.

Phase studies led one investigator^{3, 4, 6, 7}, to regard as probable the following course of reaction in normal Portland cement paste, insofar as it can be predicted from studies made on iron-free systems, and with limitation to the reactions related to the disposition of the alumina: Initial formation of solid $\text{Ca}(\text{OH})_2$ and high-sulphate sulphoaluminate $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$ in equilibrium with the gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$; depletion of the gypsum and its disappearance as a solid phase; diminution of the concentration of SO_3 and eventual formation of a solid-solution phase probably having initially about the composition of the low-sulphate sulphoaluminate $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$; continued formation of the latter phase at the expense of the high-sulphate sulphoaluminate which eventually disappears; progressive change in the composition of the low-sulphate phase through solid solution with calcium-aluminate hydrate. This sequence assumes that metastable solid solution forms in preference to stable $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$.

The mechanism of the retardation of setting by means of gypsum is not well established. Recent data^{9, 10} on rates of heat evolution add materially to the observed facts.

The need exists for new methods of investigating the cement-water system. The method of differential thermal analysis¹¹, which has shown promise, is described briefly in the following.

Results of Differential Thermal Analysis.

In the method of differential thermal analysis the difference in temperature between adjacent samples of experimental and reference material is continuously recorded under conditions of uniformly rising furnace temperature. Records were obtained (using ignited Al_2O_3 as the reference material) for nine commercial Portland cement clinkers hydrated for periods up to nine months with and without gypsum. Curves thus obtained were compared with curves obtained using $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 31\text{H}_2\text{O}$, members of the solid-solution series between $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$ and $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Ca}(\text{OH})_2 \cdot 12\text{H}_2\text{O}$, and certain other preparations, mostly gels. On the basis of this method the investigators¹¹ reached the following, among other, conclusions.

The calcium-sulphoaluminate, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 31\text{H}_2\text{O}$, appears to form as a metastable phase only during the first few days in most of the cements studied. The solid solution $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Ca}(x)\text{SO}_4 \cdot (1-x)(\text{OH})_2 \cdot (13 \text{ to } 12)\text{H}_2\text{O}$ also appears to form as a metastable phase in some cements of high ratio of alumina to iron, but not until all the gypsum has combined. The stable sulphate phase may be the product characterised by an endothermic bulge at 130 deg. to 140 deg. C. There is

some indication that it may be bearing solid containing $\text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$. The isometric tricalcium-aluminate hexahydrate does not form in hydrating cements although it apparently sometimes forms in hydrating clinkers.

The authors of the bulletin reserve judgment on the conclusiveness of the method, but the procedure appeals to them as simple and capable of giving strong indications under conditions which make many other methods useless.

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The Cement Industry in Great Britain.

THE final report of the census of production carried out by the Board of Trade in the cement industry has now been published (Final Report on the Census of Production for 1948, Vol. 1, Trade J, Cement. H.M. Stationery Office. Price 1s. 6d. net).

Figures are in some cases given for the years 1948, 1937 and 1935, but these are not comparable because whereas the figures for the year 1948 relate to Great Britain only those for the earlier years relate to the United Kingdom including Northern Ireland. On this basis, some of the figures given are as follows. Value of total production of cement: 1948, £27,628,000; 1937, £11,707,00; 1935, £9,135,000. Cost of materials and fuel used: 1948, £15,520,000 1937, £5,445,000; 1935, £3,693,000. Net value of output per person employed: 1948, £1,188; 1937, £513; 1935, £572. The production of calcareous cement was 8,479,000 tons in 1948, 7,527,000 tons in 1937, and 5,936,000 tons in 1935. The production of high-alumina cement in 1948 was 55,000 tons valued at £588,000; no figures are given for previous years.

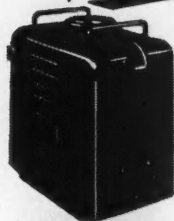
Exports of cement from the larger establishments totalled £2,651,000 in the year 1948.

The fuel and power consumed comprised the following. Coal: 2,363,800 tons at a cost of £6,306,000 in 1948; 2,236,400 tons at a cost of £2,031,000 in 1937; and 1,937,400 tons at a cost of £1,364,000 in 1935. Oil fuel: 757,000 gallons at a cost of £35,000 in 1948. Electricity: 623,851,000 kw.-hours at a cost of £1,976,000 in 1948; 590,593,000 kw.-hours at a cost of £822,000 in 1937; 407,322 kw.-hours at a cost of £563,000 in 1935.

The number of people employed in the industry was 10,191 in 1948, 12,211 in 1937, and 9,508 in 1935. The wages and salaries paid in the year 1948 amounted to £3,947,000.

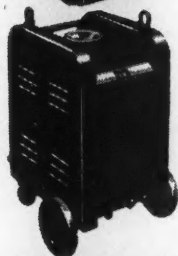
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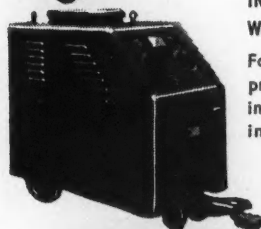
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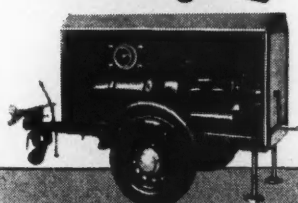
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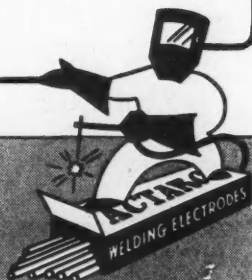
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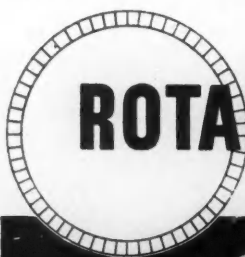
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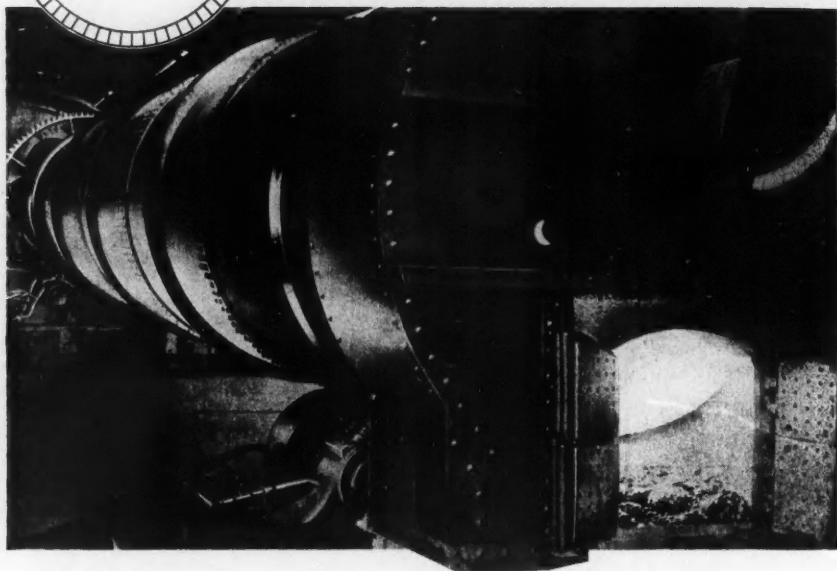
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Hydration of Cement at Medium Temperatures.

IN this journal for May, 1951, the hydrothermal reactions were considered for cement hydrated at atmospheric temperatures, that is, up to 30 deg. C., and from 100 deg. C. to, in some cases, 1000 deg. C., and it was stated that hitherto little had been reported of work relating to temperatures between 30 deg. C. and 100 deg. C. Mr. G. L. Kalousek and Mr. M. Adams have now published in the Journal of the American Concrete Institute for September, 1951, the results of their research on "Hydration Products Formed in Cement Pastes at 25 to 175 deg. C."

The tests, in which differential thermal analyses were used, indicated the chemical nature of a possibly new hydration product, called phase X, which may be responsible for the high strength of steam-cured cements. The following is a summary of the results of the tests.

Thermal analysis indicated that the products containing R_2O_3 and SO_3 formed and transformed in cements hydrating at temperatures of 25 deg. to 100 deg. C. in the sequence $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O$ and presumably the analogous sulphoferrates appeared first, but were converted to the related solid solutions of these products which, in turn, were converted to phase X. At 70 deg. to 100 deg. C. the solid solution was found in greater abundance than the trisulphate product, but transformed rapidly to phase X. Pure $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O$ did not decompose at temperatures below 100 deg. C., but between 100 deg. and 105 deg. C. this compound broke down into $CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O$ and gypsum, and between 130 deg. and 140 deg. C. into $3CaO \cdot Al_2O_3 \cdot 6H_2O$ and gypsum.

At processing temperatures of 80 deg. to 100 deg. C. the products showed a marked decrease in the endothermic bulge between 130 deg. and 140 deg. C., characterising phase X. This phenomenon could indicate a change in porosity of phase X or an alteration of its chemical composition. Phase X may be a gel consisting of all the oxide constituents of cement, and could not be prepared from raw mixtures not containing CaO , Fe_2O_3 , or SiO_2 .

The hydrogarnets in pure condition showed pronounced dehydration bulges. The hydrogarnet forming initially from $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ transformed slowly to the related compound or compounds, occurring in the form of hexagonal crystals, and a gel. Hydrogarnets were not detected in cement pastes hydrated at temperatures between 25 deg. and 175 deg. C.

Finely-ground silica (quartz) reacted with cement at 175 deg. C. to form the orthorhombic dicalcium-silicate hydrate described by Thorvaldson and Shelton.¹ About one-third to a half of the reaction product of cement and 8 per cent. to 10 per cent. of silica consisted of dicalcium-silicate hydrate, the remainder being the still unidentified product or products responsible for the strength of the hydrated cement. This mixture gave the product of the lowest strength. The product from a mixture containing 40 per cent. to 45 per cent. of finely-ground

¹ T. THORVALDSON and G. R. SHELTON. Canadian Journal of Research, Vol. 20 (1936), page 20.

silica had the greatest strength, which was about three times as great as that of the cement without the addition of silica. The composition of the cementitious phase with this amount of silica added seems to approach a molar ratio ($\text{CaO}:\text{SiO}_2$) of unity, which phase showed an extensive exothermic peak at $840 (\pm 5)$ deg. C.

Cement Works in Haiti.

It is reported that a concession granting the exclusive rights for the manufacture of cement in Haiti has been granted to the Italcementi Fabrice Riunite Cemento Company, of Bergamo, Italy. The concession is for an initial period of 25 years and is to be exploited by a company registered as Le Ciment d'Haiti, S.A., with a registered capital of 300,000 dollars. Provision is made that this company shall construct, within a period of two years, one or more factories for the manufacture of Portland cement. The company will be allowed to import equipment free of duty and will be permitted, in respect of each factory, to operate for a period of five years without payment of tax. The cement to be produced is to be sold at a price to be agreed, but which will be at least 20 per cent. below that of imported cement.

Determination of Free Magnesia in Portland Cement.

A method which is claimed to be an improvement on Bogue's method of determining the free magnesia content of Portland cement was put forward by Mr. K. Miyazawa and Mr. M. Amemiya at last year's convention in Tokyo of the Cement Engineering Association of Japan.

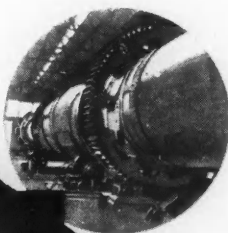
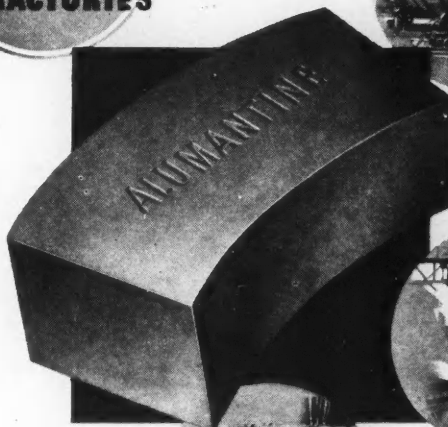
The modification of Bogue's method is as follows: (1) Substitution of ethyl alcohol for methyl alcohol to shorten the time for testing and to make the end point clearer; (2) Addition of a small quantity of oxalic acid (to shorten the time required for the determination); (3) Substitution of glycerin by ethylene glycol to make filtration easier; (4) Fine grinding of sample (to pass No. 325 sieve) and rubbing the sample with alcohol in an agate mortar.

A New Cement Works in North Africa.

THE new cement works of the Société des Ciments Artificiels d'Oranie, at Oran, North Africa, was described in this journal for November, 1951. Construction commenced in December, 1946 (not 1949, as stated in the article), and by the middle of the third quarter of 1949 the civil engineering work was completed. At the end of the same quarter the installation of the machinery which proceeded during the erection of the buildings was nearly finished. Testing proceeded during the fourth quarter of 1949, and the works was officially put into operation at the beginning of 1950. By December 31, 1950, the amount of cement produced was 150,000 metric tons. These data are from "Revue des Matériaux de Construction" for July, 1951.

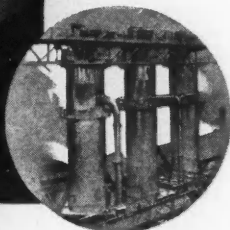
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HIGH ALUMINA REFRACTORIES



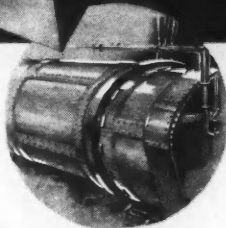
40-45% ALUMINA

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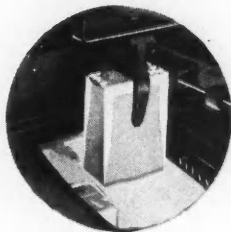
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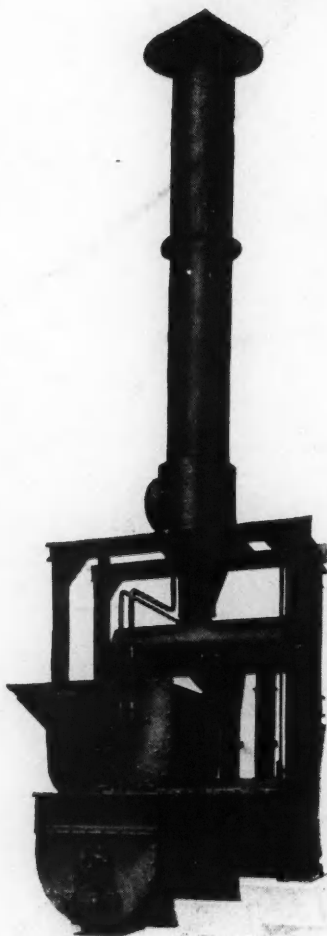


Illustration shows general view of Schulthess Hydrator from feed end

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- *Minimum dust*
- *Rapid slaking under ideal conditions*
- *Continuous operation*
- *Automatic and continuous heating of slaking water*
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